



Published in final edited form as:

*Ann Occup Hyg.* 2016 November ; 60(9): 1072–1083. doi:10.1093/annhyg/mew053.

## A New Miniature Respirable Sampler for In-mask Sampling: Part 1— Particle Size Selection Performance

Peter Stacey<sup>1,\*</sup>, Andrew Thorpe<sup>1</sup>, Rhiannon Mogridge<sup>1</sup>, Taekhee Lee<sup>2</sup>, and Martin Harper<sup>2</sup>

<sup>1</sup>Health and Safety Executive, Science Division, Health and Safety Laboratory, Harpur Hill, Buxton SK17 9JN, United Kingdom

<sup>2</sup>Exposure Assessment Branch, Health Effects Laboratory Division, National Institute for Occupational Safety and Health, Centers for Disease Control and Prevention, Morgantown, WV 26505, USA

### Abstract

The Health and Safety Laboratory has developed a miniature respirable sampler to gain a better understanding of the exposure of workers to hazardous substances when they are wearing respiratory protective equipment (RPE) or helmets with visors in the workplace. The study was in two parts and the first part, described herein, was to develop the sampler and test its collection characteristics. Assessment of the impact of the sampler on RPE safety and its comparability with traditional laboratory-based approaches to measure protection factors was discussed in a second article. The miniature sampler (weight—5.4 g, length—13 mm) was designed to fit into the space available between the nose and chin of an individual inside a filtering facepiece type mask and has a radially omnidirectional inlet with a porous foam particle selector that allows the collection of the respirable fraction on a downstream filter. The sampling efficiency was compared with the respirable convention. A close match with the respirable convention was obtained at a flow rate of 1 l min<sup>-1</sup> and the 50% penetration cut off value ( $d_{50}$ ) was 4.08  $\mu$ m. After 3 hours sampling in high humidity (95%), the penetration curve had shifted towards smaller particle sizes ( $d_{50}$  = 3.81  $\mu$ m) with 88% of the calculated bias values within 10%. The miniature sampler measured respirable dust and crystalline silica mass concentrations comparable with performance of the Safety In Mines Personal Dust Sampler (SIMPEDS), commonly used in Great Britain, at a flow rate of 0.8 l min<sup>-1</sup>. The  $d_{50}$  for the miniature sampler at 0.8 l min<sup>-1</sup> (4.4  $\mu$ m) is within 5% of the  $d_{50}$  of the SIMPEDS at its prescribed flow rate of 2.2 l min<sup>-1</sup> (4.2  $\mu$ m). These results indicated that the miniature sampler was a good candidate to proceed with tests with RPE described in the second part of this series of two papers.

\* Author to whom correspondence should be addressed. Tel: +01298 218554; peter.stacey@hsl.gsi.gov.uk.

**Publisher's Disclaimer:** This publication and the work it describes were funded by the Health and Safety Executive (HSE) Exposome Strategic Research Programme and the National Institute for Occupational Safety and Health. Its contents, including any opinions and/or conclusions expressed, are those of the authors alone and do not necessarily reflect HSE policy or the official position of the US Centres for Disease Control and Prevention/the Agency for Toxic Substances and Disease Registry.

Supplementary Data: Supplementary data can be found at <http://annhyg.oxfordjournals.org/>.

## Keywords

crystalline silica; filtering facepiece; inward leakage; mask; miniature; respirable; respiratory protective equipment; RPE; sampler

---

## Introduction

It is a principle in good occupational hygiene practice that respiratory protective equipment (RPE) is only employed to protect the worker when engineering controls are not effective or are impractical to employ (HSE, 2002). There are many processes, especially when working with powered tools and materials containing crystalline silica, where levels of hazardous material remain high despite following principles of good practice and RPE is needed to control exposures to below workplace exposure limits. The respirable fraction of airborne dust is measured when assessing materials that are hazardous when deposited in the gas-exchange or alveolar region of the lung, e.g. respirable crystalline silica in the case of exposure to crystalline silica dust. Respirable dust measurements are usually carried out by pulling air through a device that separates particles according to their aerodynamic size and traps the respirable airborne dust on a filter. The respirable size fraction is defined by the International Organisation for Standardisation/Comité Européen de Normalisation/American Conference of Government Industrial Hygienists (ISO/CEN/ACGIH) according to a curve describing the probability of penetration to the alveolar region of particles that are mostly  $<16\text{ }\mu\text{m}$  and a 50% cut of aerodynamic diameter of  $4.0\text{ }\mu\text{m}$  (CEN, 1993), which is the particle size for which the penetration of particles is 50%.

Basic face filtering particulate (FFP) disposable dust masks (designated types FFP 2 or 3 in Europe, or N95 and N99 in USA) are often used by workers exposed to mineral dust. These dust masks are usually manufactured from felted spun or melt blown fibres, and sometimes have a rubber seal and/or clip to compress the area over the nose. In Europe, the laboratory test performance requirements for these masks can be used to derive nominal protection factors (NPF), of 12 for type FFP 2 or 50 for type FFP 3 (CEN, 2001). This implies that for every 12 (FFP 2), or 50 (FFP 3), particles outside the mask only 1 particle should be permitted to pass. Several studies examining the protection offered by some masks in the workplace have shown that a laboratory-based performance test is not an accurate assessment of how masks really perform in the workplace as there are many differences in conditions between the two environments, e.g. work-rates, duration, training, supervision, and use with other work equipment and protective equipment (Morre and Smith, 1978; Myers and Peach, 1983; Wallis *et al.*, 1993; Howie *et al.*, 1996). An assigned protection factor (APF) is applied to reflect the likely protection achieved in the workplace and is set at the level of respiratory protection that is realistically achieved by 95% of adequately trained and supervised wearers using properly functioning and fitted equipment (CEN, 2005). FFP 2 or 3 type masks are not expected to perform as well in the workplace as suggested by their calculated NPF and are therefore APFs of 10 and 20 in the UK, respectively. These APFs are mostly based on expert expectations, theory, and limited workplace protection factor evidence. The shortage of workplace evidence to confirm the APFs has an impact when assessing a worker's exposure with RPE. A major issue is how to assess the 'true' exposure

of a worker involved in a health study when wearing a mask, since the sampler to evaluate exposure is placed outside the protective equipment. An assumed correction, based on the APF for the mask an individual is wearing, can be applied to the airborne challenge concentration obtained from a sampler in the worker's breathing zone outside of the mask. This correction reduces the challenge concentration value and assumes that the mask produces at least this reduction, which may not be the case. Protection factors derived from laboratory tests or inadequately simulated workplace situations may be overly optimistic. Ideally, assessment of the protection offered by RPE needs to be informed by measurements made in real workplaces during real RPE use (Howie, 2005).

This work describes the development and performance of a miniature respirable size selective sampler designed to fit within a filtering facepiece type mask. A previous study (Lee *et al.*, 2005) described a system that could be used in both laboratory and field studies which used particle counting devices that were strapped to a manikin or worker to derive total inward leakage by comparing measurements from outside the mask with those measured inside. Their system utilizes a probe within the mask to transport particles to the particle counter *via* a length of tubing. Particle counters offer a very sensitive and almost real-time measurement, although they generally select a limited particle size and their additional size and weight is potentially cumbersome for workers during routine work. However, compliance for exposure assessment for most respiratory hazards is determined by mass, so count data may not always clearly represent the risk. It is therefore advantageous to develop methods to assess the airborne mass concentration of hazardous particles an individual is exposed to inside a mask when breathing. Sampling and collecting particles inside the mask avoids potential sample losses from particles adhering to the plastic tubes that lead to other devices, such as the particle counter. In addition, particles from other sources (mask, face, and mouth) or generated within the respiratory system or by talking might also be counted by the particle counter as if they had originated from outside. The filter from an in-mask sampler can collect the analyte of interest *in situ* and the result of that analysis can potentially be used to determine a protection factor that is specific to a type of hazardous material, if the analytical technique is sensitive enough. It is important that the miniature sampler has a performance that is comparable to the respirable sampler, commonly used for workplace measurement in Great Britain (GB), if it is to be used in workplace protection factor studies, to ensure that measurements are comparable to historic exposure monitoring results and to inform data contributing to exposure modelling.

## Design and Manufacture of the Miniature Sampler

The available space, between the nose and the chin on an individual, inside a filtering facepiece type mask was measured using modelling clay to fill the volume. A miniature sampler manufactured from aluminium was designed (Fig. 1) to occupy the 1.5 cm<sup>3</sup> space that was available when measured on a clay model from a single assessment of a male individual. The sampler is 13 mm in length when measured from its flat top to its base. It was intended that sampler would seal with its base pressed flat against the mask material (Fig. 2). The connection to the pump projects through the mask and is sealed tight against the mask using a push nut fitting commonly used in RPE fit testing (TSI Inc., Shoreview, MN, USA). The miniature sampler was designed to be as light as possible (5.4 g) to prevent any

additional weight compromising the fit of the mask to the face of the worker. A radially omnidirectional annular inlet (~1 mm annular spacing) was incorporated into the design so that particles following the expected leakage path, along the contours of the face, might be collected efficiently. Broadly similar inlet designs are used in some in-mask probes for inward leakage laboratory tests. Several trial prototype samplers were assessed to ensure the sampler was robust enough to cope with any interaction that may occur with the worker during sampling. The contact between the sampler and the filter was sealed using two fat 'O' rings that are the same as those used in the commercially available SWINNEX filter holder that has previously been used as the basis for an inhalable dust sampler (Lidén and Surakka, 2009). A plug of polyurethane foam [PUF; 90 pores per linear inch (PPI)] was used to collect and remove larger particles allowing only the respirable particle fraction to be collected on the filter. This foam plug was placed directly behind the annular inlet and before the filter. The foam plug was located in the axial part downstream of the annular inlet. The filter was a 13-mm diameter disc of polyvinylchloride (PVC) material with a pore size of 5 µm. The base and inlet sections of the sampler are secured together with a screw thread.

## Method and Materials

### Particle size selection tests in laboratory conditions

The performance of the miniature sampler was tested using a calm air chamber at the Health and Safety Laboratory (Fig. 3). This chamber has been described by Stacey and Thorpe (2010), and Stacey *et al.*, (2014). The design of the test system was based on that described by Lidén and Kenny (1991) used for the measurement of aerosol penetration through cyclone samplers. The approach requires measurements of the aerodynamic size distribution of an aerosol penetrating through a sampler under test and that of the aerosol challenging it. The two size distributions are compared to obtain the particle penetration characteristics of the sampler.

A poly-disperse aerosol of glass ballotini beads (Spherglass 5000 CP00 Potters, Europe) was generated in the calm air chamber using a Palas model 1000 rotating brush generator (RBG, GmbH, Karlsruhe, Germany). The charge level on the aerosol was reduced using an ionising air blower positioned directly beneath the rotating brush generator shown in Fig. 3. The particle size distribution of aerosol drawn through the porous foam sampler was analysed using an Aerodynamic Particle Sizer (APS, model 3321, TSI Inc.) and compared to aerosol drawn through an identical set of tubing without a porous foam plug. The latter was measured as the challenge aerosol. The airborne concentration was kept within 50–100 particles cm<sup>3</sup>, to minimize particle counting errors within the APS.

The porous foam sampler was characterized over a range of flow rates from 1 to 2 l min<sup>-1</sup> using the following procedure. Samples of 1-minute duration were drawn through each system in turn, with 1-minute intervals between samples. In each case, 3 reference and 2 foam penetration samples were taken for each measurement. Three repeat measurements were made at each flow rate. The reference and foam sampler particle concentrations were averaged at each particle size, and foam penetration measured as a fraction of the reference aerosol using a spreadsheet. These data were transferred to a curve-fitting computer program (Tablecurve 2D, SPSS Systat Software Inc., CA, USA) where the calculated fractional

penetration was normalized to 1 at 1  $\mu\text{m}$  to eliminate effects caused by non-linearity of the APS inlet below this size (Kenny and Lidén, 1991). The measured penetration curves over the respirable particle size range were compared with the respirable curve defined in EN 481 (CEN, 1993). The particle size at which 50% of particles penetrate ( $d_{50}$ ) was calculated from the fitted curves at each flow rate to assess its closeness with the  $d_{50}$  cut off value referred to in EN 481 (CEN, 1993). In addition, the measured performance data for the miniature sampler at each flow rate was assessed against the respirable target convention defined in EN 481 (CEN, 1993), using the bias map approach described in EN 13205:2 (CEN, 2014). The predicted bias between the fitted performance curve and the target convention was calculated for an array of challenge size distributions, described in EN 13205:2 (CEN, 2014). To confirm comparability of  $d_{50}$  values with the Safety In Mines Personal Dust Sampler (SIMPEDS), the performance of the miniature sampler with a foam insert was retested at flow rates of 1.1 and 0.8  $\text{l min}^{-1}$ . The particle size penetration measurement at these flow rates was based on an average of 3 reference and 3 foam penetration samples of 2-minute durations.

### Particle size selection tests in high humidity

A worker's exhaled breath is likely to cause a highly humid environment [ $>90\%$  relative humidity (RH)] within a mask because it contains moisture. This may affect the penetration characteristics of the foam separator since the foam may become saturated preventing particles from penetrating through. To assess if the miniature sampler and foam separator were able to cope with high humidity air, the calm air chamber was modified to incorporate a steam generator (Fig. 3). The air was recirculated so that the water vapour introduced into the test area could be maintained at a high level ( $\sim 95\%$  RH), so that the miniature sampler was challenged simultaneously to both high humidity and particles. Particle penetration tests were performed at a flow rate of 1  $\text{l min}^{-1}$  (determined from the previous penetration tests) before and after exposure to particles and high humidity. The temperature and RH inside the chamber were measured with a Logit 12 bit data logger (DCP Micro developments Ltd, Great Ellingham, UK). The RH for the tests in laboratory conditions was 47%, which was then increased to assess the performance of the foam in high-moisture conditions. During periods of high humidity ( $\sim 95\%$ ), the air was drawn through the sampler at 1  $\text{l min}^{-1}$  using a personal air sampling pump rather than the APS to prevent possible damage to the instrument. During the penetration tests, the steam generator was switched off and the calm air chamber was switched to filtered mode rather than recirculating, and the RH was allowed to fall below 90% so that it did not affect the APS readings (the APS specification (TSI Inc.) states that it can be used in RHs up to 90%). Penetration tests were carried out as soon as possible thereafter so that the foam did not dry out, although this was unlikely since the humidity inside the chamber remained high. Throughout the tests, the aerosol inside the test chamber was found to be extremely stable with very little temporal fluctuation in particle concentration. The sampler flow rate was checked before and after each test using a primary calibrator (model 4046/4116 TSI Inc.) and was found to be within 1–3% of the target value. The  $d_{50}$  cut off particle size value was determined with sampling time to assess if moisture loading would affect the foam penetration. A significant lowering in  $d_{50}$  would mean that the foam retained too many particles resulting in fewer particles being collected on the back up filter (Kenny *et al.*, 2001).

## Mass Concentration Comparison Tests

Sampling efficiency tests are important to assess if candidate samplers meet the standard sampling convention defined in EN 481 (CEN, 1993). However, differences may still occur between different samplers due to wall deposition of particles in cassettes, (HSE, 2014; Soo *et al.*, 2014) leakage between vortex chamber and cassette in cyclone samplers, variation in performance with dust loading and dust loss from filters. It is important to know if any potential differences are caused by these other factors, in addition to the particle selection performance. There is also a need to ensure that the performance of the miniature sampler is comparable with the sampler most commonly employed to take the measurements on the outside of the mask to compare exposure data. Therefore, the performance of the miniature sampler was checked by comparing the measured air concentration with that of the SIMPEDS (Casella Ltd, Bedford, UK) which is frequently used as the standard respirable size selective sampler in the GB. Tests comparing the mass concentrations measured by each sampler were conducted using aerosols of coal dust (ground Pittsburgh coal; Lee *et al.*, 2012) and Arizona road dust (ARD, ISO 12103 PT 1. A1 Ultrafine) generated at the National Institute for Occupational Safety and Health (NIOSH) in the USA. When aerosolized, these powders have aerodynamic particle sizes distributions within the respirable size range and median diameters of about 4.5  $\mu\text{m}$  for the coal dust (Lee *et al.*, 2012) and 2.8  $\mu\text{m}$  for the ARD (Stacey *et al.*, 2014). The calm air chamber at NIOSH was used for these tests and is documented by Lee *et al.*, (2012). The test system comprised an aluminium cylinder about 0.7 m high with 0.5 m diameter. The aerosol was generated using a fluidized bed aerosol generator (Model 3400, TSI Inc.). The aerosol was introduced into the chamber through a  $^{85}\text{Kr}$  aerosol neutralizer (Model 3012A, TSI Inc.). The aerosol entered the chamber in a radial direction to help achieve a uniformly distributed aerosol inside of the chamber. The particle concentration inside the chamber was monitored at the sampling point using an APS (Model 3321, TSI Inc.). The SIMPEDS was operated with 25 mm diameter, 5  $\mu\text{m}$  pore size, PVC filters at 2.2  $\text{l min}^{-1}$ . The miniature sampler was operated at both 0.8 and 1  $\text{l min}^{-1}$ . A change to a slightly lower flow rate was made to compensate for initial low airborne respirable dust concentration results when compared with the SIMPEDS. The filters were weighed on micro balance (XP6U, Mettler-Toledo, Columbus, OH, USA; readability 0.1  $\mu\text{g}$ ). Filters were passed through an electrostatic bar (Mettler-Toledo) to dissipate static charge. A single measurement for each filter was made after allowing for balance stabilization. The average uncertainty of measuring 28, 13-mm diameter PVC filters with loadings from 6 to 135  $\mu\text{g}$  was  $\pm 1 \mu\text{g}$ . The mass concentrations of respirable dust collected by the miniature sampler were compared with those collected by the SIMPEDS. The relationships between paired mass concentration values were compared graphically and trend lines were set to zero when the intercept was not significantly different.

The mass concentration of respirable crystalline silica (RCS) in the ARD dust collected by the miniature sampler was measured using method based on NIOSH method 7500 (NIOSH, 2003). These results were compared with those of samples collected with the SIMPEDS sampler using a direct on-filter analysis method MDHS 101 (HSE, 2005).



The calm air chamber at HSL (Fig. 3) was used to perform an additional test to challenge the miniature sampler, operating at a flow rate of  $0.8 \text{ l min}^{-1}$ , to aerosols from a simulated work task. The task involved the cutting of sandstone (containing 90% quartz) with a powered saw. The chamber has been used for other respirable sampler mass concentration comparison studies (Stacey and Thorpe 2010; Stacey *et al.*, 2014). The power saw was located in the top of the chamber and operated from outside through gloved ports. The paired samplers (2 miniature and 2 SIMPEDS) were located (within  $\sim 10 \text{ cm}$ ) at the bottom of the chamber. All the sampling was carried out in calm air conditions ( $<0.5 \text{ m s}^{-1}$ ). A series of 10 different dust concentrations were generated with sampling times between 9 and 13 min. Filters were conditioned in a room with controlled humidity ( $50 \pm 5\%$ ) and temperature  $20 \pm 2^\circ\text{C}$ . They were weighed using an UX6 ultra-microbalance (Mettler Toledo Ltd) with the readability set to  $0.1 \mu\text{g}$ . Each filter was weighed three times before and after sampling and an average sampled mass was reported. Each dust concentration was calculated by dividing the weight gained by the volume of air sampled. The respirable dust concentrations were compared with the theoretical ideal 1:1 relationship collected by the SIMPEDS. The 95% confidence interval of the slope was calculated to assess if the relationship between the two samplers was significantly different from an ideal relationship value of 1.00.

## Results

### Comparison with the respirable convention

The relationship between flow rate and measured  $d_{50}$  is shown in Fig. 4. The  $d_{50}$  of the penetration curves measured using glass beads decreased as the flow rate was increased. A flow rate of  $1 \pm 0.05 \text{ l min}^{-1}$  gave a  $d_{50}$  close to  $4 \mu\text{m}$  ( $d_{50}$  of 4.08 with a standard deviation from 3 tests of  $<1\%$ ). Figure 5 compares the penetration curves of the miniature sampler with the respirable curve at a flow rate of  $1 \text{ l min}^{-1}$  when tested in normal laboratory conditions with a RH of 47% and after 3 h of sampling a 95% RH atmosphere. The sampler maintained a  $d_{50}$  within 5% of the target value of  $4 \mu\text{m}$  after 3 h of accumulated sampling in an environment of about 95% humidity. Table 1 shows the  $d_{50}$  obtained from three repeat tests before and at hourly intervals in 95% RH. The prolonged exposure at high humidity shifted the particle selection curve slightly towards lower values ( $d_{50} = 3.81$ ). The bias maps showing the predicted average bias over the whole respirable range when sampling dusts with a range of mass median aerodynamic diameters (MMAD) and geometric standard deviations are provided for the miniature sampler when sampling a  $1 \text{ l min}^{-1}$  in 47% RH and after sampling of 3 h in 95% RH (Fig. 6). At a flow rate of  $1 \text{ l min}^{-1}$  and 47% RH, 97% of the predicted biases were within  $\pm 10\%$  for all the particle size distributions considered by EN13205:2 for a respirable sampler (CEN, 2014). The proportion of predicted bias values lying within 10% after 3 h sampling in 95% RH was lower (88%).

### Comparability with the SIMPEDS respirable sampler

Results for the tests carried out at NIOSH to compare dust concentration measurements of the miniature sampler with the SIMPEDS when sampling ARD and coal dust are shown in Fig. 7. The sampling times for ARD were between 30 – 120 minutes and respirable dust mass range measured on filters from the miniature sampler was from 30 –  $500 \mu\text{g}$ . The

miniature sampler underestimated the respirable mass concentrations compared with the SIMPEDS at the flow rate of  $1 \text{ l min}^{-1}$ , so it was reduced to  $0.8 \text{ l min}^{-1}$  to obtain a closer match. The slope of the trend line with the miniature sampler at the reduced flow rate when sampling ARD was 0.82 (95 % confidence interval was 0.73 – 0.89).

The sampling times for the coal dust sampling were between 20 – 75 min and the mass range of respirable dust collected on the filter inside the miniature sampler was from 45 – 200  $\mu\text{g}$ . The intercept of the trend line (plotted excluding the two highest points) with the intercept set to zero ( $y = 0.87x$ ) was significantly different from zero so the trend line is plotted with an intercept (Fig. 7). Two sets of paired respirable coal dust samples at the highest mass concentrations recorded low values for the miniature sampler when compared with the SIMPEDS. The 95% confidence interval for the trend line slope for the remaining paired values comparing the respirable coal dust concentrations was from 0.89 – 1.28, which includes the ratio of 1.

The trend line relationship comparing respirable dust concentrations collected by the SIMPEDS and miniature sampler during the simulated work task with sandstone is  $y = 0.98x$  (Figure 8). The 95% confidence interval of the slope was 0.87–1.10 which includes the value of 1.00 in its range; however, coefficient of determination was smaller than for the other trend lines ( $r^2 = 0.71$ ) indicating a larger variability of paired values from the trend line relationship. The mass of deposit loaded on these samples was between 37 and 110  $\mu\text{g}$ .

Figure 9 compares the concentration of RCS measured on filters collected by the miniature and SIMPEDS samplers. The equation for the relationship between the miniature sampler and the SIMPEDS is  $y = 0.93x$  and the 95% confidence interval of the slope coefficient (0.85–1.01) includes the ideal value of 1.00 when the intercept was set to zero. The mass of RCS measured in the ARD was from 30 to 282  $\mu\text{g}$ . Measurements of RCS for the coal dust samples are not compared as results for the miniature sampler are close to the X-ray diffraction limit of detection.

The particle size selection performance of the miniature sampler at the reduced flow rate of  $0.8 \text{ l min}^{-1}$  was measured and its  $d_{50}$  cut off value for 50% penetration was found to be 4.4, which is within 5% of the SIMPEDS  $d_{50}$  of 4.2 (Stacey *et al.*, 2014). The particle size penetration curves for the measurement of a single SIMPEDS operating at  $2.2 \text{ l min}^{-1}$  and the miniature sampler operating at  $0.8 \text{ l min}^{-1}$  are compared in Fig. S1 (see Supplementary Fig. S1, available at *Annals of Occupational Hygiene* online).

## Discussion

Foam separators can potentially change their particle selection characteristics because they act as a filter of larger particles and become loaded with dust. As the particle loading increases the effective size of the pores may decrease resulting in a more effective filter which moves the particle selection towards smaller sizes and a subsequent reduction in  $d_{50}$  (Kenny *et al.*, 2001). High humidity environments ( $\sim 94\% \text{ RH}$ ) might be expected to have a similar effect as moisture in the air condensates inside the foam and restricts the cell size. Slightly lower values for the  $d_{50}$  were observed after 3 h of sampling in humid air and a



higher proportion of predicted sampler bias values were >10%. This suggests that the foam may need replacement at suitable intervals to ensure optimum particle selection performance is maintained during sampling in high humidity conditions.

It has been demonstrated that generally, there is a good correspondence between respirable dust concentrations, RCS concentrations and  $d_{50}$  values obtained with the miniature sampler operating at a flow rate of  $0.8 \text{ l min}^{-1}$  and the SIMPEDS cyclone. The slope of the trend line relationship between concentrations measured with the miniature and SIMPEDS sampler for respirable dust when challenged to ARD was found to be 0.81; indicating that on average the miniature sampler may still sample less than the SIMPEDS with this grade of mineral dust, at the reduced flow rate. However, the slope value for ARD is comparable with that found for other work. Stacey *et al.*, (2014) obtained slope values of 0.82 and 0.80 for the IOM 'multidust' sampler, (another sampler that uses foam to separate the respirable fraction) compared with the SIMPEDS, when exposed to aerosols of ultrafine and medium grades of ARD, respectively. The difference is smaller for RCS analysis in the filters containing ARD. In this article, the paired RCS concentration values for ultrafine grade ARD using XRD show agreement with the SIMPEDS (average RCS measurement difference is within 7% of the SIMPEDS). This suggests that the difference in respirable dust concentrations of ~19 % might be due to the performance of the different analytical approaches (i.e. gravimetric and XRD analysis) rather than the samplers' different sampling efficiencies. Intensity of an XRD measurement can be affected by particle size issues if the measured sample comprises a significant number of larger or smaller particles than the calibration dust. However, XRD is less sensitive to particle size differences than infrared spectroscopy (Stacey *et al.*, 2009) and the ARD MMAD ( $\sim 2.8 \mu\text{m}$ ) was similar to in the calibration dust after aerosolisation ( $1.9 \mu\text{m}$ ). The reason for the difference between the two techniques is not clear, although a simpler explanation is probably that the RCS analyses did not include samples above  $300 \mu\text{g}$  which may have influenced the slope value.

Some of the higher paired concentration values indicate that the miniature sampler may under sample respirable dust with higher mass loadings even though it has been shown that foam selectors can be tolerant to loading. Kenny *et al.*, (2001) investigated the limitations on loading with the PUF foam separator used in the IOM 'multidust' sampler and demonstrated that the  $d_{50}$  cut off value did not substantially change even with foam loadings up to  $\sim 15 \text{ mg}$  from both mineral processing and foundry workplace and laboratory-generated samples. There are two additional possible practical explanations for the reduced mass concentration from the higher dust concentration samples (i) a reduced flow rate caused by increased back pressure as the mass of dust collected on the air sample filter increases and (ii) losses of dust from higher loaded filters during handling when removing them from the sampler. The removal of the filter from the miniature sampler can be very difficult if the filter remains lodged in the thread of the inlet section and doesn't remain on top of the filter support. Hence, dust can be lost when the filter flexes during the efforts to retrieve it. The internal fittings (e.g. seal types and support) may have to be slightly modified to rectify this problem. Most mass loadings tested in this work ( $30\text{--}500 \mu\text{g}$ ) far exceed the loading range expected behind a mask ( $<5 \mu\text{g}$ ), which is the intended application of the miniature sampler, although further work will be needed to investigate the performance limitations of the miniature sampler if used for routine workplace aerosol sampling. The next phase of the work will

compare the performance of the miniature sampler with the techniques used in laboratory-based protection factor studies for RPE and the safety of the device in volunteer studies (Mogridge *et al.*, 2016). This will also provide the opportunity to further compare the collection performance of the miniature sampler with currently accepted quantitative performance assessment methods for inward leakage in RPE.

A further stage of this project will assess the capabilities of analytical techniques to obtain a hazard specific measurement within the expected mass ranges inside a mask. The miniature sampler will be useful for in-mask sampling purposes where analytical techniques exist that have sensitivity comparable with a particle counter or flame photometer. Such techniques currently exist for flour dust allergens which can measure down to about 10 ng; however, current analytical methods for RCS are not able to achieve sensitivity down to low ng quantities with current limits of detection of about 5–10 µg.

## Conclusion

The Health and Safety Laboratory has developed a miniature respirable size selective sampler that has the potential to offer occupational hygienists the opportunity to collect relevant dust samples behind masks or visors, (where the expected concentration is low), to more accurately assess the ‘true’ exposure risk of workers and to obtain more relevant health-related measurements and protection factors.

The sampler bias, particle size penetration curve and 50% penetration values are comparable with the respirable convention at a flow rate of 1 l min<sup>-1</sup>. The majority (94%) of calculated bias values for the miniature sampler were within 10% of the respirable convention for a range of theoretical particle size distributions.

To obtain a closer match with the performance of the SIMPEDS sampler, which is routinely used by HSL for workplace studies, the miniature sampler should be run at 0.80 l min<sup>-1</sup>.

## Acknowledgments

The authors gratefully acknowledge the assistance of Dr Vaughan from the Health and Safety Laboratory (HSL), and Drs Echt and Cauda from the National Institute for Occupational Safety and Health who provided editorial and technical reviews. The authors also acknowledge the contributions of Mr M Merrill (HSL) who provided computer aided design drawings and Mr M Johnson in the workshops at HSL who manufactured the prototypes.

## References

- CEN. EN 481 Centre European de Normalisation Workplace atmospheres: Size fraction definitions for measurement of airborne particles in the workplace, European Committee for Standardisation, rue de Stassart. Vol. 36. B-1050 Brussels, Belgium: 1993.
- CEN. EN 149, Respiratory protective devices — Filtering half masks to protect against particles — Requirements, testing, marking European Committee for Standardisation, rue de Stassart. Vol. 36. B-1050 Brussels, Belgium: 2001.
- CEN. EN 529 Respiratory protective devices — Recommendations for selection, use, care and maintenance — Guidance document B-1050 European Committee for Standardisation, rue de Stassart. Vol. 36. B-1050 Brussels, Belgium: 2005.
- CEN. Brussels, Belgium: Laboratory performance test based on determination of sampling efficiency European Committee for Standardisation, rue de Stassart. Vol. 36. B-1050 Brussels, Belgium: 2014.

- EN 13205-2, Workplace exposure Assessment of sampler performance for measurement of airborne particle concentrations.
- Howie, M., Johnstone, J., Weston, P., et al. Workplace effectiveness of respiratory protective equipment for asbestos removal work HSE Contract Research Report. Vol. 112. London: HMSO; 1996.
- Howie RM. Respiratory protective equipment. *Occup Environ Med.* 2005; 62:423–8. 362. [PubMed: 15901892]
- HSE. The Control of Substances Hazardous to Health Regulations (as amended 2002). Norwich, United Kingdom: The Stationery Office; 2002. <http://www.hse.gov.uk/pubns/priced/15.pdf>
- HSE. MDHS 101 Methods for the determination of hazardous substances Crystalline silica in respirable airborne dusts: direct-on-filter analyses by infrared spectroscopy and X-ray diffraction. London: HSE Books; 2005.
- HSE. MDHS 14 Methods for the determination of hazardous substances, General methods for sampling and gravimetric analysis of respirable, thoracic and inhalable aerosols. HSE Books; 2014. Available at <http://www.hse.gov.uk/pubns/mdhs/pdfs/mdhs14-4.pdf> [Accessed 30 August 2016]
- Kenny LC, Lidén G. A technique for assessing size selective dust samplers using the APS and polydisperse test aerosols. *J Aerosol Sci.* 1991; 22:91–100.
- Kenny L, Chung K, Dilworth M, et al. Applications of low-cost, dual-fraction dust samplers. *Ann Occup Hyg.* 2001; 45(1):35–42. [PubMed: 11137697]
- Lee S, Grinshpun S, Adhikari A, et al. Laboratory and field evaluation of a new personal sampling system for assessing the protection provided by the N95 filtering face-piece respirators against particles. *Ann occup Hyg.* 2005; 40(3):245–57.
- Lee T, Lee EG, Kim S, et al. Quartz measurement in coal dust with high-flow rate samplers: laboratory study. *Ann Occup Hyg.* 2012; 56(4):413–25. [PubMed: 22186376]
- Lidén G, Kenny LC. Comparison of measured respirable dust sampler penetration curves with sampling conventions. *Ann Occup Hyg.* 1991; 35:485–504. [PubMed: 1746808]
- Lidén G, Surakka J. A headset-mounted mini sampler for measuring exposure to welding aerosol in the breathing zone. *Ann Occup Hyg.* 2009; 53(2):99–116. [PubMed: 19196747]
- Mogridge R, Stacey P, Forder J. A new miniature respirable sampler for in-mask sampling: Part 2—Tests performed inside the mask. *Ann Occup Hyg.* 2016
- Morre D, Smith T. Measurement of protection factors of chemical cartridge, half-mask respirators under working conditions in a copper smelter. *Am Ind Hyg Assoc J.* 1978; 37:453–8.
- Myers W, Peach M. Performance measurements on a powered air-purifying respirator made during actual field use in a silica bagging operation. *Ann Occup Hyg.* 1983; 27:251–9. [PubMed: 6314865]
- NIOSH. Silica, Crystalline, by XRD (filter redeposition) Method 7500. Atlanta, Georgia: United States Department of Human Health and Human Sciences, National Institute for Occupational Safety and Health; 2003. Manual of Analytical Methods (NMAM). Available at <http://niosh.cihcsp.com/pdfs/7500.pdf>
- Soo JC, Lee T, Kashon M, et al. Coal dust deposit on internal surface of respirable size selective samplers. *J Occup and Env Hyg.* 2014; 11:D215–9. [PubMed: 25204985]
- Stacey, P. Evaluation of HSE report 112/1996 Workplace effectiveness of respiratory protective equipment for asbestos removal work IR/MF/98/03. Harpur Hill, Buxton SK179JN, UK: The Health and Safety Laboratory; 2003.
- Stacey P, Kauffer E, Moulut JC, et al. An International comparison of the crystallinity of calibration materials for the analysis of respirable alpha-quartz using X-ray diffraction and a comparison with results from the infrared KBr disc method. *Ann Occup Hyg.* 2009; 53:639–49. [PubMed: 19531809]
- Stacey, P., Thorpe, A. Testing of high flow rate respirable samplers to assess the technical feasibility of measuring 0.05 mg m<sup>-1</sup> respirable crystalline silica The Health and Safety Executive, Research Report 825. Health and Safety Executive; 2010. Available at <http://www.hse.gov.uk/research/rpdf/r825.pdf> [Accessed August 2016]
- Stacey, P., Mecchia, M., Verpaele, S., et al. Differences Between Samplers for Respirable Dust and the Analysis of Quartz—An International Study, Silica and Associated Respirable Mineral Particles.

Harper, M., Lee, T., editors. ASTM International; West Conshohocken, PA: 2013. p. 73-102.STP 15652013

Stacey P, Lee T, Thorpe A, et al. Collection efficiencies of high flow rate personal respirable samplers when measuring Arizona road dust and analysis of quartz by x-ray diffraction. *Ann Occup Hyg.* 2014; 58:512–23. [PubMed: 24470535]

Wallis G, Menke R, Chelton C. Workplace testing of a disposable negative pressure half-mask dust respirator (3M 8710). *Am Ind Hyg Assoc J.* 1993; 54(10):576–83.

Author Manuscript

Author Manuscript

Author Manuscript

Author Manuscript

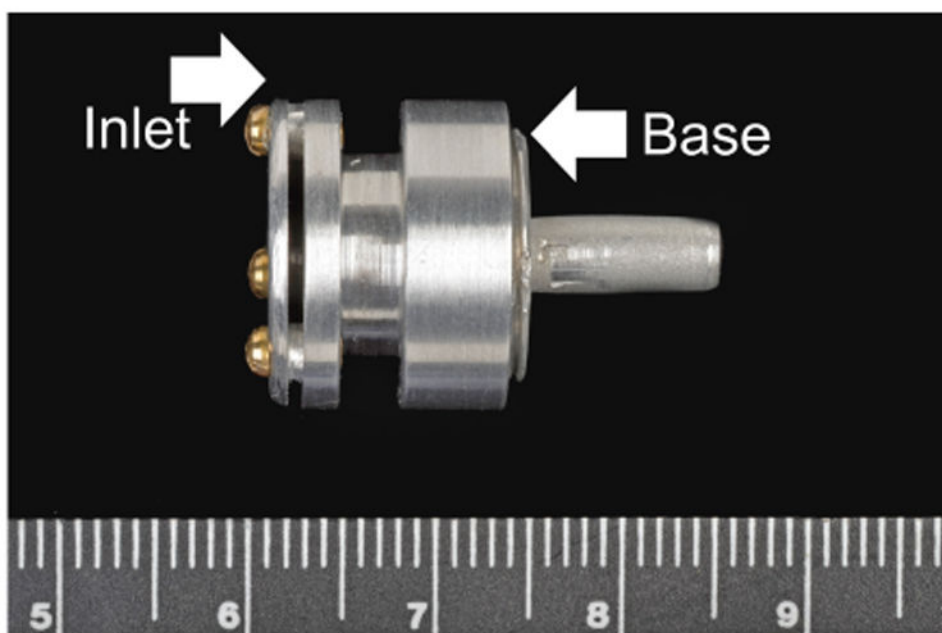


Figure 1. Photograph of the miniature sampler with centimetre scale



**Figure 2. A miniature sampler fitted to a filtering facepiece (FFP 3) mask**



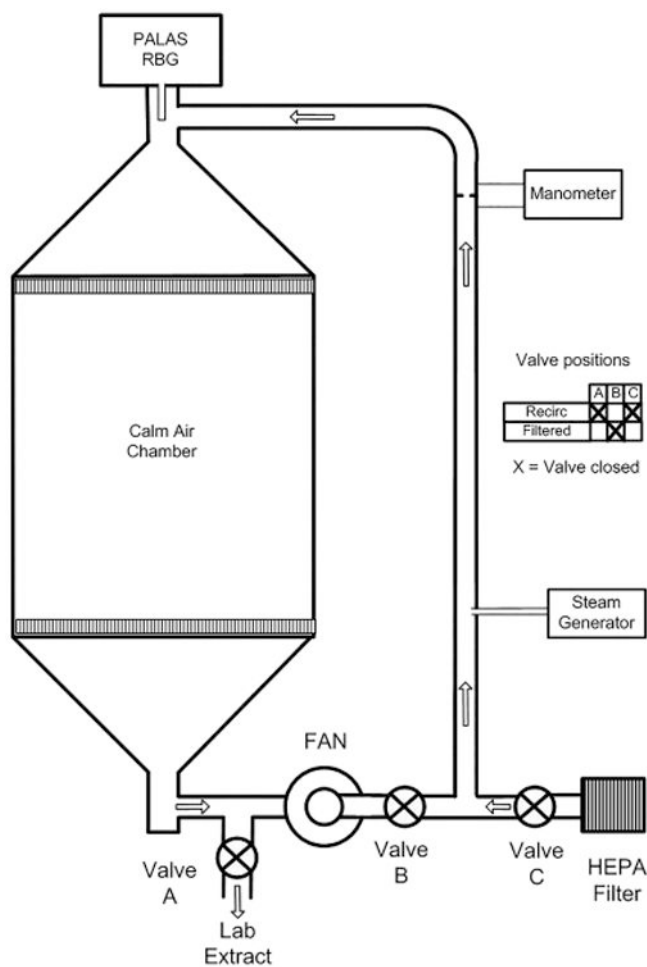
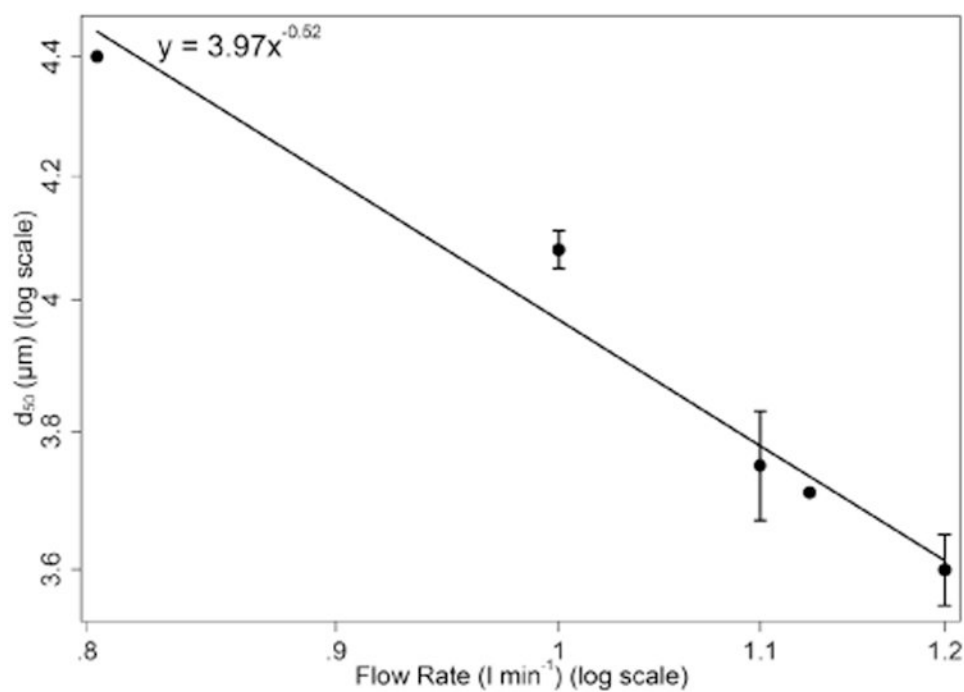
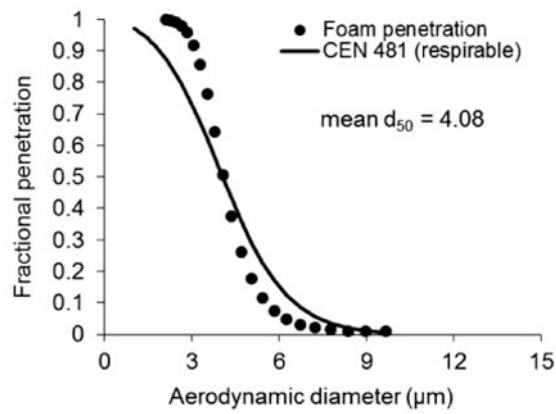


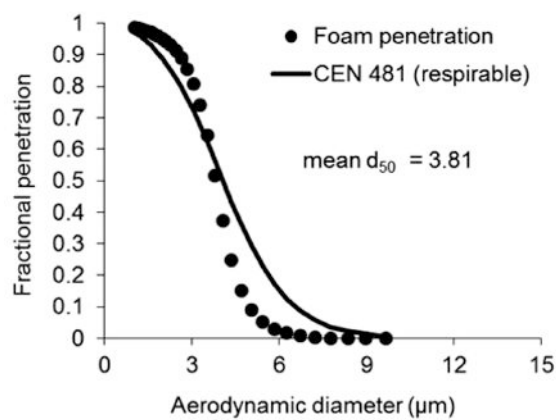
Figure 3. Schematic diagram of the calm air humidifying chamber at HSL



**Figure 4.** The relationship (log/log scale) between flow rate and the particle size at 50 % penetration for the miniature sampler



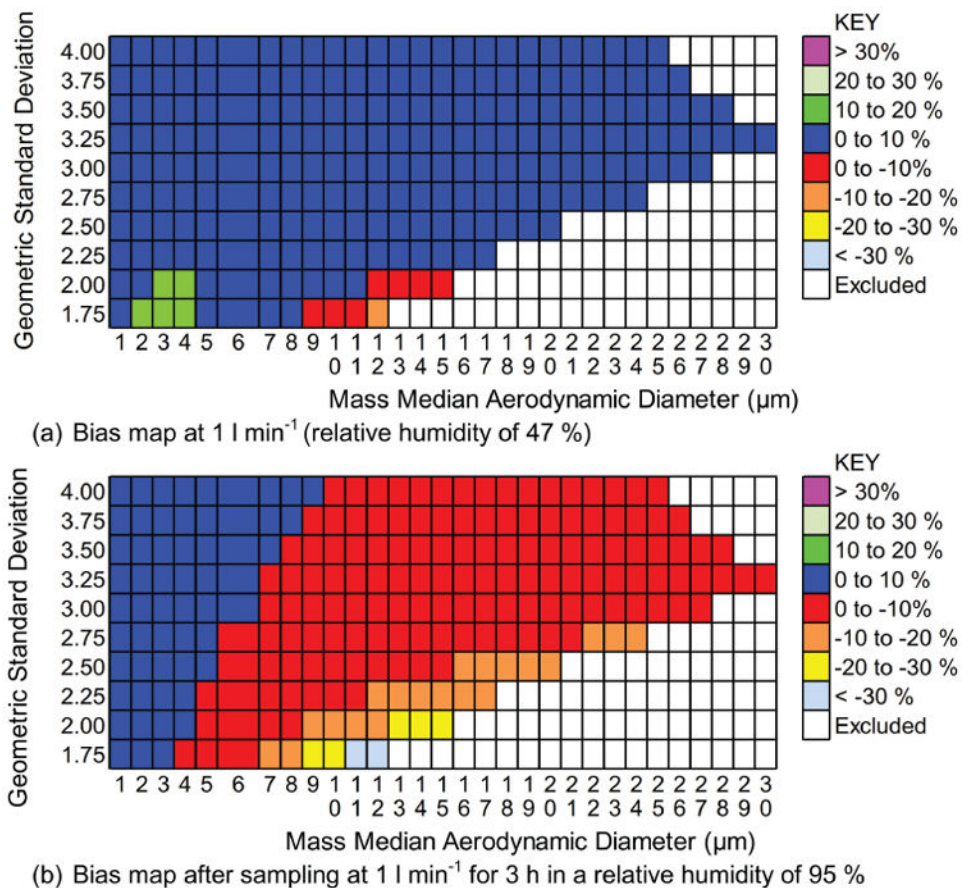
(a) At 1 l min<sup>-1</sup> and relative humidity 47 %



(b) 3 h sampling at 1 l min<sup>-1</sup> in a relative humidity of 95 %

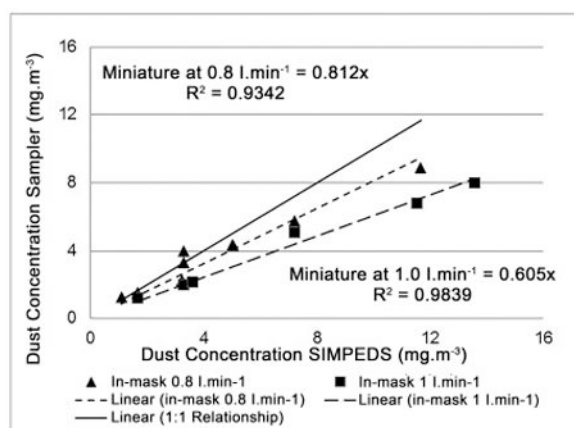
**Figure 5.**

Comparison between the penetration curves for the miniature sampler at a flow rate of 1 l min<sup>-1</sup> and the respirable convention when measured in (a) laboratory conditions of 49% RH and (b) after 3 h sampling in 95% RH.

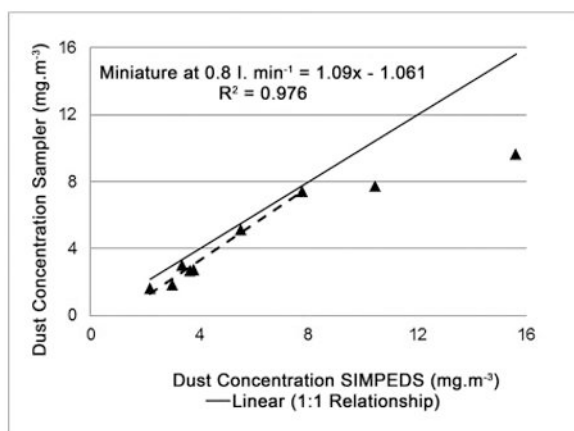


**Figure 6.**

Bias maps for the predicted overall bias when sampling a range of aerosols with different mass median aerodynamic diameters and geometric standard deviations for (a) in laboratory conditions of 47% RH and (b) after sampling for 3 h in 95% RH.



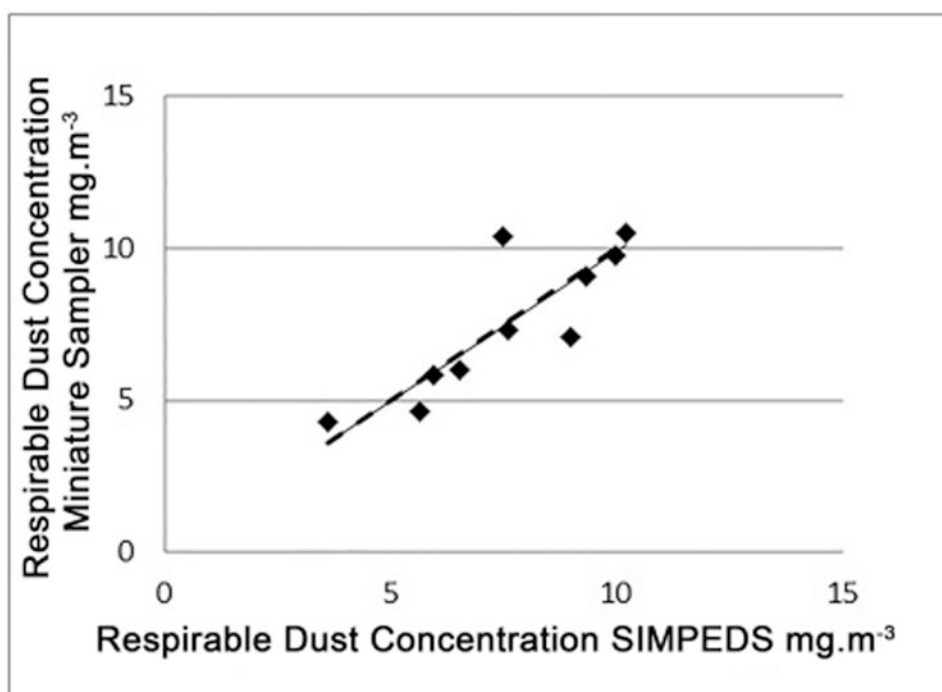
(a) Arizona Road Dust



(b) Pittsburgh Coal Dust

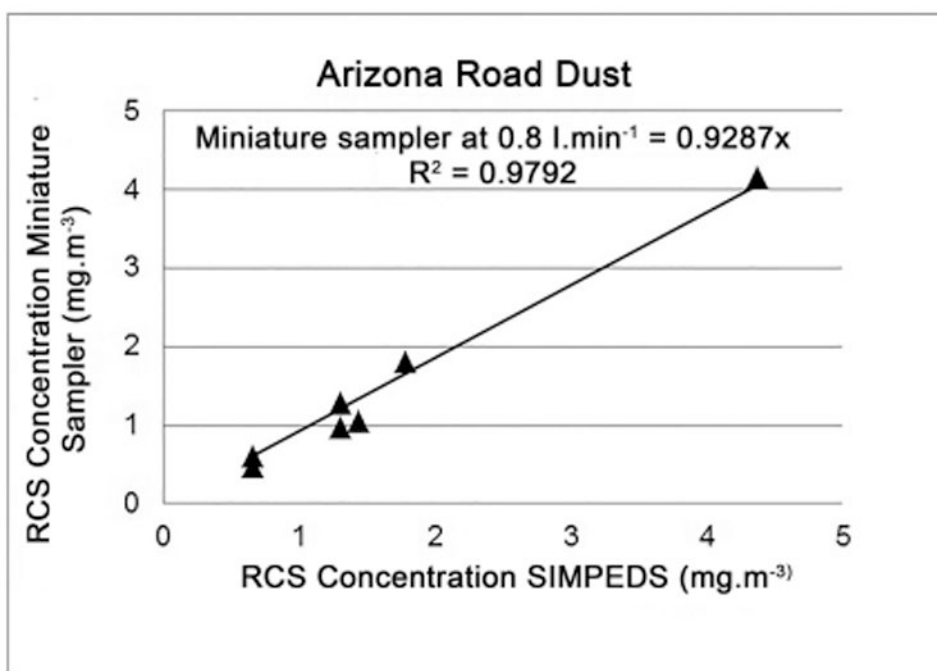
**Figure 7.**

Comparison of respirable dust concentrations for the miniature (0.8 l min<sup>-1</sup>) and SIMPEDS samplers when sampling aerosols of Arizona Road Dust and Coal Dust. The regression line for the coal dust measurements is drawn excluding the two highest points.



**Figure 8.**  
Comparison of respirable dust concentrations between the miniature sampler at  $0.8 \text{ l min}^{-1}$  and SIMPEDS samplers when exposed to aerosols from the cutting of sandstone using a Mitre saw.





**Figure 9.** Comparison of concentrations of RCS measured by miniature sampler at 0.8 l min<sup>-1</sup> and SIMPEDS sampler when sampling aerosolized ARD.

**Table 1**  
**Results of  $d_{50}$  measurements from miniature sampler sampling efficiency tests carried out at a flow rate of  $1 \text{ l min}^{-1}$  and exposed to high RH**

Test conditions	1	2	3
	~47% RH	After 1 h @ 94.5% RH	After 3 h @ 94.8% RH
Mean $d_{50}$ ( $\mu\text{m}$ )	4.17	3.92	3.81
Standard deviation ( $\mu\text{m}$ )	0.05	0.02	0.1
Per cent difference from target $d_{50}$ of 4	+4%	-2%	-5%